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ALTERNATIVE METHOD FOR THE EVALUATION OF FUSED GLASS-TO-METAL SEALS

General Electric Company

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F30602-78-C-0086

ROME AIR DEVELOPMENT CENTER
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Glass-to-metal seals on electronic component packages make it possible to obtain electrical feed-thru while maintaining hermeticity throughout severe environmental exposure. As a result of this study, it was determined that sealed sub-assemblies will generally pass the least severe thermal shock requirement of MIL-STD-883, but that the same (previously accepted) lots will exhibit a 6 percent, or higher, failure rate as a result of manufacturing stresses imposed by cover welding, delidding, and thermal bakes or soaks. During the course of this study, the use of tighter controls on the more critical glass

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sealing processes was shown to reduce the above reject rate to less than 1 percent. Success in matched glass-seal making depends on the use of three thermal treatments for Kovar, de-carburizing, pre-oxidation and sealing. The present work deals specifically with the use of pre-oxidation and sealing. An oxide film on Kovar, prior to sealing promotes glass wetting and provides for the development of a chemical-mechanical bond at the glass-metal interface.

A determination for residual intergranular oxide is useful for judging the quality of a matched glass-to-metal seal where pre-oxidation is a requisite for sealing. Residual oxides can be detected by metallographic techniques, and the range of 2.0 - 6.5 μm is appropriate for high quality glass seals. Other effects are also detectable by the evaluation of metallographic cross-sections of sealed packages. These include volume fraction of bubble formation, bubble size, bubble distribution, glass-metal separation and other interface criteria.

EVALUATION

This effort supports RADC TPO R5B, Solid State Device Reliability. Appendix 4 of this report is a test method in the format of MIL-STD-883, Test Methods and Procedures for Microelectronics. The method, titled "A Metallographic Test for Glass-to-Metal Seal Quality," has been submitted to the Preparing Activity (PA) of MIL-STD-883, for possible inclusion in the document. During the course of the study, the use of tighter controls on residual intergranular oxide and gas bubble distribution and size required by the test method reduced package reject rates from 6% to less than 1%. Package lots that fail the requirement of the test method are acceptable if they pass fine and gross leak test after thermal shock from +150°C to -65°C for 15 cycles.

John E. McCormick

JOHN E. McCORMICK
Project Engineer

ALTERNATIVE METHOD FOR THE EVALUATION OF FUSED GLASS-TO-METAL SEALS

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1.0 INTRODUCTION

Glass-to-metal seals on IC lead frames, transistor headers and other electronic component packages make it possible to obtain electrical feed-thru while maintaining hermeticity throughout severe environmental exposure such as that required by MIL-STD-883. In the most exacting applications, the hermeticity requirement is most consistently met by the use of the so-called matched seal. Many of these seals use ASTM Alloy F15 (Kovar) and a glass with matching coefficient of expansion. In practice, industry specialists do the glass-seal work and the component fabricator uses the purchased part to complete a hermetic assembly. Despite the widespread, and long-standing development of glass-seal technology, the industry seems plagued with component failure due to loss of hermeticity.

As a result of the present study, it was determined that sealed subassemblies will generally pass the least severe (Method 1011.2 Cond. A) thermal shock requirement of MIL-STD-883, but that the same (previously accepted) lots will exhibit a 6%, or higher, failure rate as a result of manufacturing stresses imposed by cover welding, de-lidding, and thermal bakes or soaks. This failure rate is very costly with today's complex circuits and packages. During the course of this study, the use of tighter controls on the more critical glass sealing processes was shown to reduce the above reject rate to less than 1%.

1.1 Thermal Treatments. Success in matched glass-seal making depends on the use of three thermal treatments for Kovar, including de-carburizing, pre-oxidation and sealing. The present work deals specifically with the use of pre-oxidation and sealing. An oxide film on Kovar, prior to sealing, promotes glass wetting and provides for the development of a chemical-mechanical bond at the glass-metal interface. During the course of this work, several lots of selectively-oxidized headers were fabricated and tested for hermeticity. In addition, a number of failure analyses were made on components obtained over the same period of time, and a correlation was made with purchased components that were tested according to the metallographic requirements developed during this program.

As a result, an improvement in quality can be related directly to improved controls in the sealing process. During this program we have emphasized the need for control of the pre-oxidation step in the glass-sealing process. It appears that pre-oxidation is a more difficult process to control than has been realized by fabricators. Also, the effect of oxidation on an alloy such as ASTM F15 is not too well understood by fabricators of sealed headers or by users of sealed packages.

It is important to understand that, if pre-oxidation is part of the sealing process, both under-oxidation and over-oxidation must be controlled. Too little oxide appears to cause lack of bonding (leakers), and too much oxide causes leakers as well as problems with lead integrity. This study was made to determine the minimum-maximum limits required to avoid each type of problem.

The determination of oxide composition and its effect on sealing with different glasses, under differing conditions is beyond the scope of the present project, and we have concerned ourselves mainly with morphology. R. P. Abendroth[1] in his detailed study of oxidation kinetics, however, was unable to correlate mechanisms and structures by metallography. He does suggest that a minimum and maximum oxide thickness are required for good adherence, and that a thickness equivalent of 0.6 to 1.10 mg/cm^2 of oxide seems preferable for good seals. Assuming a density of 5.18 gm/cm^3 for the oxide (Fe_3O_4), the thickness suggested by Abendroth is 1.16 to 2.12μm. This is in the detectable range microscopically, but approaching the limit of optical measuring capability.

2.0 PROCEDURE

A test method based on the metallographic evaluation of the glass-to-metal seals can offer a feasible approach to measuring seal quality. The evaluation is based on the fact that: (1) the interface at the seal is formed during the period when the glass flows, (2) any following processes do not affect the seal interface, and (3) the visual appearance of this interface can be related to the condition of the metal members and the glass beads prior to the time when the glass was flowed, i.e., a relationship exists between the total oxide formed during pre-oxidation, the residual intergranular oxide remaining at the seal interface after sealing, and any tendency toward local saturation of the glass by the oxide.

In addition to the fabrication processes mentioned, a number of other factors will influence glass-seal quality. These include design variations, glass composition, glass-make-up (tube, powder pre-form, etc.), pre-cleaning, parts storage, lead and eyelet alloy composition, raw materials, pre-treatment, handling after pre-oxidation, size of lots, type of firing furnace, and so on. Each of these contributors will have some effect on glass-to-metal seal quality and on the inspection criteria developed during this program. However, the acceptance criteria are based on an evaluation of the visual appearance of the final product and the suggested limits are such that the requirement should be achievable with tighter controls on materials and processes, and with special emphasis on the pre-oxidation process.

To evaluate the effects of pre-oxidation, ASTM Alloy F15 parts were subjected to various oxidation treatments (time, temperature, atmosphere) and the extent of oxidation was measured metallographically. Samples representing the lower, median and upper degrees of oxidation were bonded to glass, and

the quality of the resulting glass-to-metal seals was evaluated. The thickness of the residual oxide at the seal interface was measured and the depth of penetration correlated with the degree of pre-oxidation and the glass seal quality. The quantitative limits for residual oxide were then applied to production seals, and a correlation established for the minimum oxide required for tight seals and the maximum oxide allowed to avoid glass and metal integrity degradation.

3.0 OXIDATION

The oxidation of ASTM F15 (Kovar) was studied as a function of time, temperature and furnace atmosphere. Samples were oxidized in laboratory batch furnaces with an air atmosphere and in production belt furnaces with typical generated gas atmospheres. Shown in Fig. 1 is the metallographic section of a Kovar sample as oxidized at 1900°F for 12 min. in still air. This photograph is intended for the purpose of showing: (1) the general nature of the oxidation process on Kovar, and (2) the measurements that can be made to monitor oxide growth.

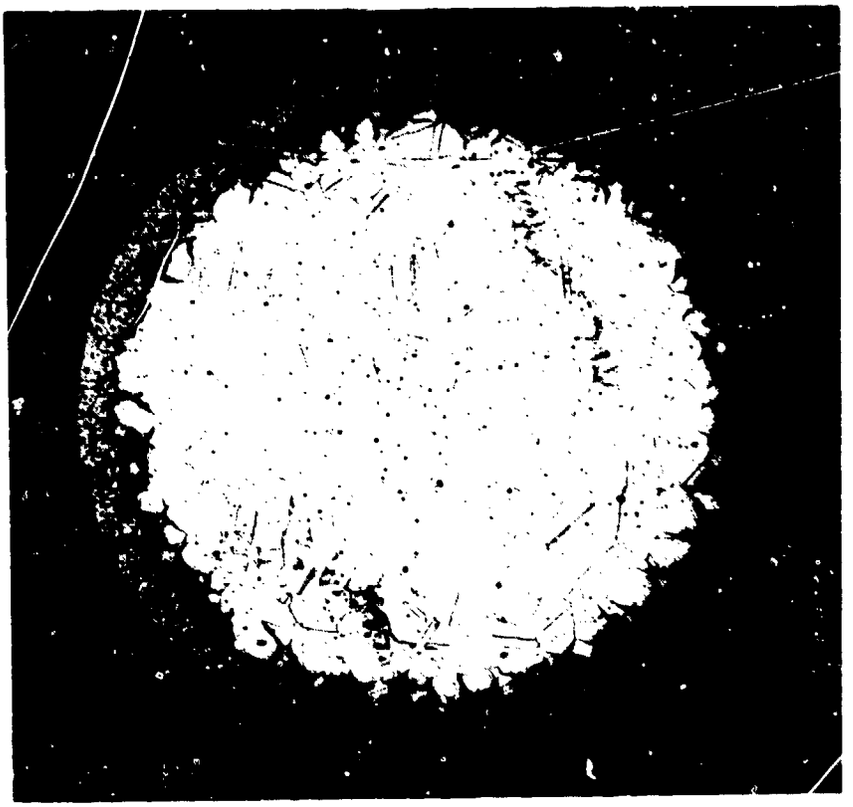


Fig. 1 Transverse Section, Oxidized Kovar Lead #106 200X 5% Nital

The section view shows an oxide scale layer (uniform gray) of 30.7μm, and intergranular oxide-metal mixture of 42.9μm under the scale layer, and the original metal microstructure (center). Note that this is as-oxidized, prior to sealing, with more oxide than would normally be used for sealing.

3.1 Oxide Growth on Kovar. A group of Kovar samples was oxidized in air over the temperature range of from 1500°F to 1900°F. Oxidizing times were varied from 3-12 minutes. Prior to oxidation, all samples were cleaned with acetone followed by an alcohol rinse.

Shown in Fig. 2 is the effect of oxidation at 1500°F; in Fig. 3, the effect at 1700°F; and in Fig. 4, the effect at 1900°F. Table I shows the measurements for oxide scale and intergranular oxide penetration taken from these samples during this series of experiments. Measurements were made by use of a filar eyepiece on a metallurgical microscope at 400X. The figure for oxide scale is taken as an average thickness over the circumference of the Kovar lead, and the measurement for intergranular oxide is taken as an average depth of penetration from the bottom of the scale layer into the metal microstructure.

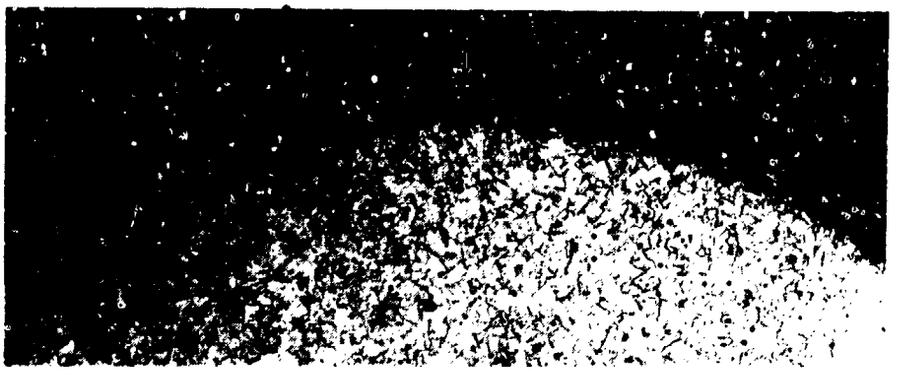
TABLE I. Oxide Growth on Kovar in Still Air

Temp °F	Time, Min	Scale Thickness μm	Intergranular Penetration μm	Ratio*
1500	3	1.2	1.7	1.4
1500	6	1.4	5.0	3.6
1500	9	1.4	6.2	4.4
1500	12	3.0	7.2	2.4
1700	3	1.4	3.5	2.5
1700	6	6.3	9.7	1.5
1700	9	9.0	13.5	1.5
1700	12	10.6	15.7	1.5
1900	3	6.5	9.0	1.4
1900	6	18.0	24.2	1.3
1900	9	24.2	41.2	1.7
1900	12	30.7	42.9	1.4

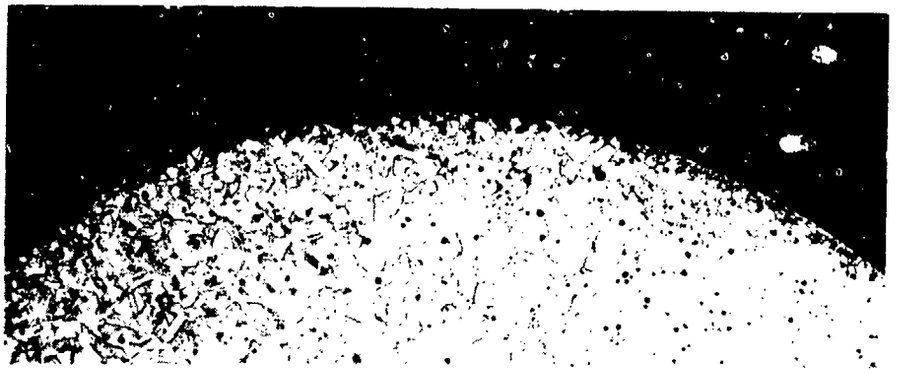
* Ratio of Intergranular Oxide to Scale Oxide

This Table shows that the ratio of intergranular oxide to oxide scale can be relatively high during the early stages of oxide formation. As total oxide content increases, the intergranular oxide-scale oxide ratio is more nearly consistent at about 1.5:1.

These data, when thickness is plotted against oxidizing time, can be represented as shown in Fig. 5.



3 min Oxidized #94 400X 5% Nital

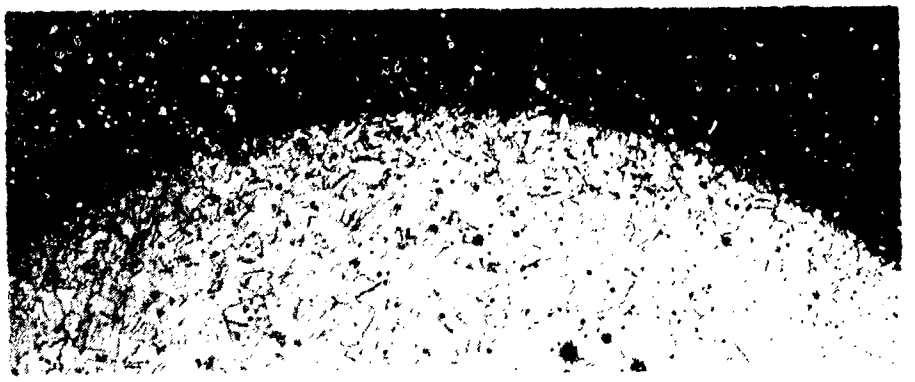


6 min Oxidized #95 400X 5% Nital

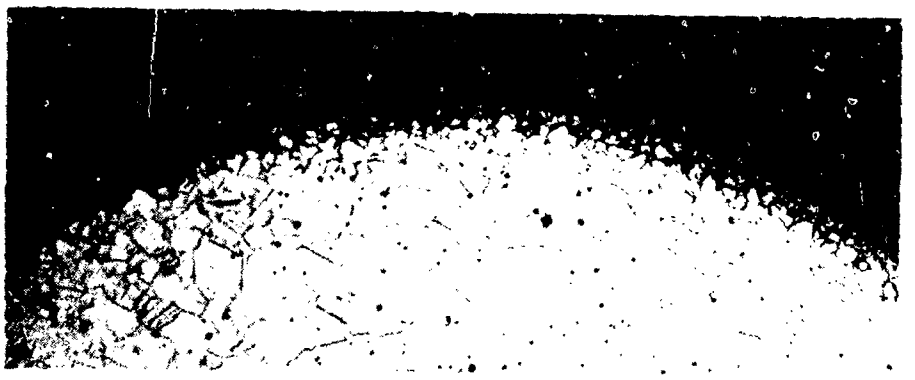
9 min Oxidized #96 400X 5% Nital

12 min Oxidized #97 400X 5% Nital

Fig. 2 Oxidation of Kovar @ 1500°F in Air



3 min Oxidized #98 400X 5% Nital

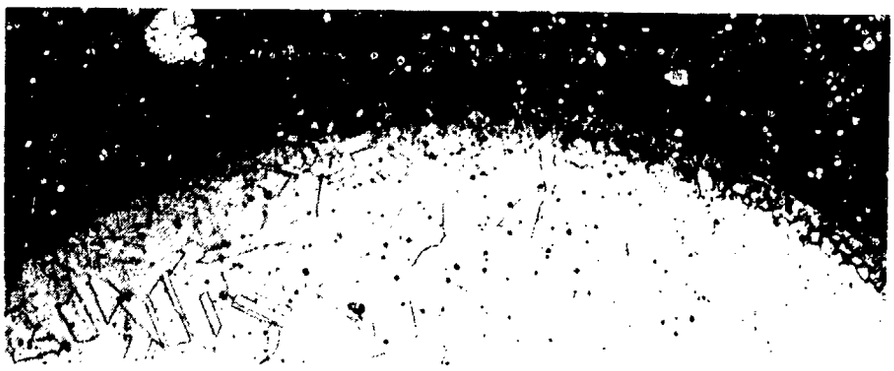


6 min Oxidized #99 400X 5% Nital

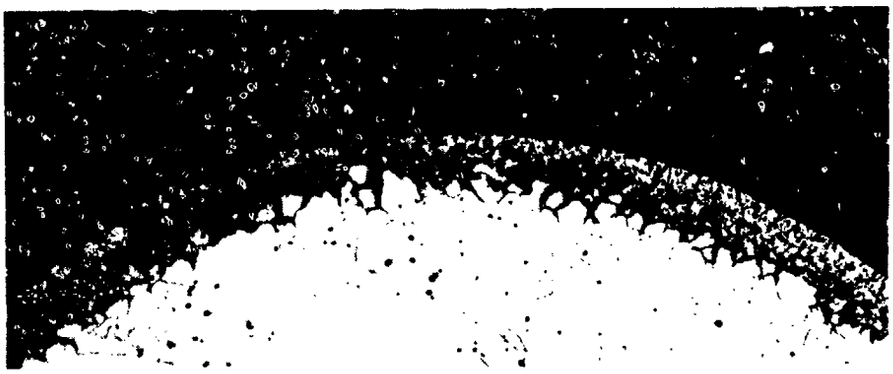
9 min Oxidized #100 400X 5% Nital

12 min Oxidized #101 400X 5% Nital

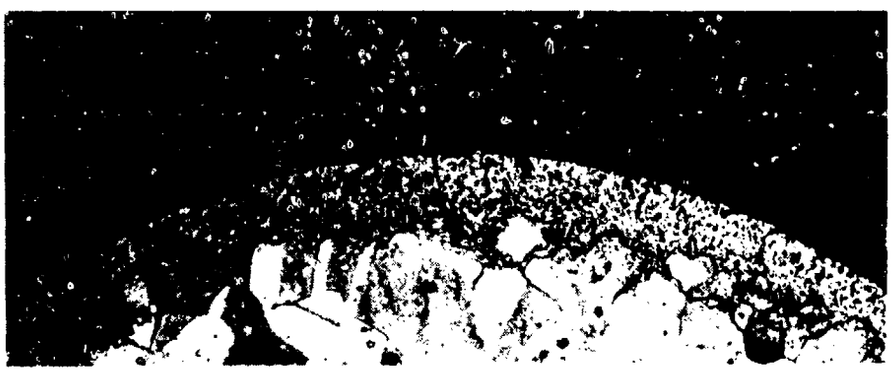
Fig. 3 Oxidation of Kovar @ 1700°F in Air



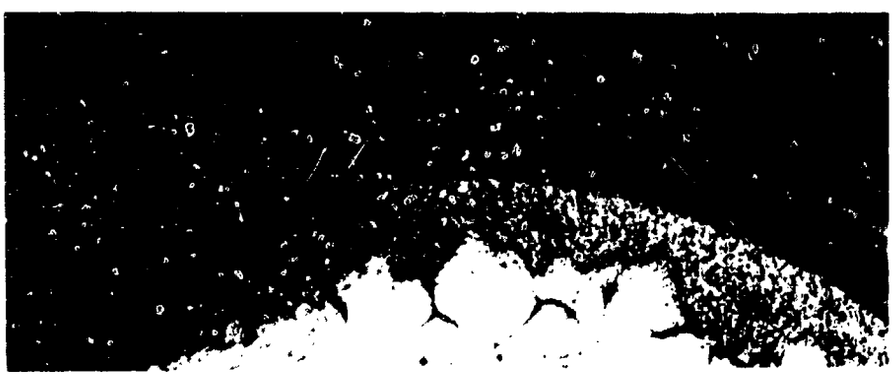
3 min Oxidized #102 400X 5% Nital



6 min Oxidized #103 400X 5% Nital



9 min Oxidized #104 400X 5% Nital



12 min Oxidized #105 400X 5% Nital

Fig. 4 Oxidation of Kovar @ 1900°F in Air

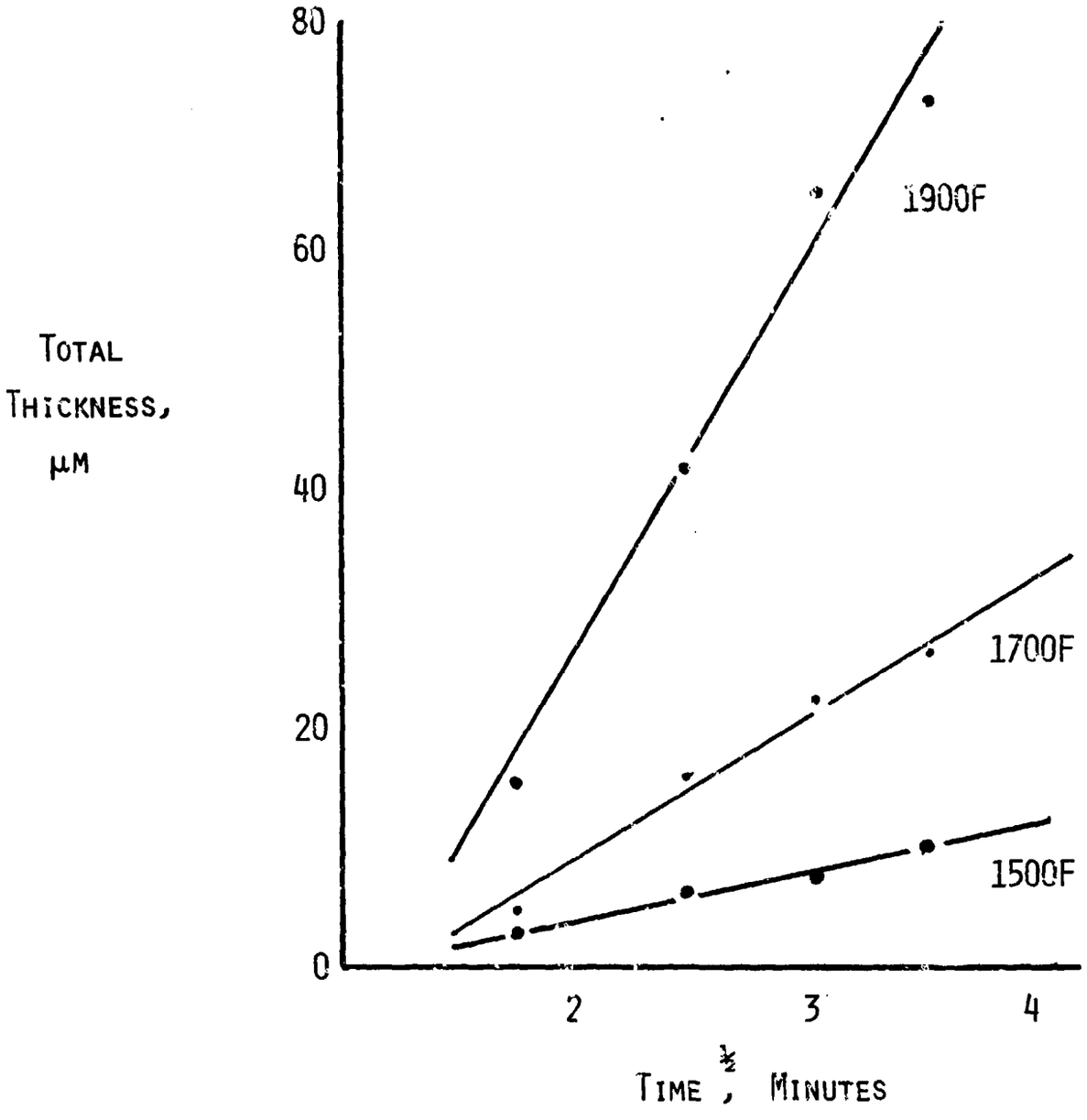


Fig. 5. Oxide Growth on Kovar in Air

These data conform, within limits of error, to the parabolic rate law. A plot of the temperature dependence of oxidation results in an activation energy of 28.1 Kcal/mole which agrees with published values for the oxidation of Kovar at from 815 to 1030°C.

3.2 Oxidation in Commercial Production Furnaces. All of the previous oxidation work had been done in laboratory furnaces using a still air atmosphere. The objective during this portion of the program was three-fold: (1) oxidize parts in a commercial furnace to compare morphology with laboratory-grown oxide, (2) develop interface criteria through metallographic and hermetic studies, and (3) determine commercial performance to specified interface criteria.

Based upon the laboratory pre-oxidation work from this study and upon earlier work[2,3] involving matched glass-to-metal seals, it was concluded that, if pre-oxidation is a requisite for sealing, a visible residual intergranular oxide should be developed, and that its presence seems to be necessary at the seal interface for consistent seal quality. Therefore, a commercial fabricator (Vendor A) was requested to supply pre-oxidized leads and eyelets to specified intergranular oxide penetration limits over the range of from 0.0 - 15.0μm. No attempt was made to dictate the use of specific furnace atmospheres since a primary objective was to evaluate present commercial practice.

Shown in Fig. 6 are the metallographic sections taken on samples from each production pre-oxidized lot. These structures show that the morphology for oxides formed in commercial furnace atmospheres is quite similar to that of oxides formed in the laboratory furnace. No further analysis of the oxides was performed, and the only directive was, in each case, a target quantity of intergranular oxide.

The oxide scale and the intergranular oxide penetration were measured on these samples in a manner similar to that described for the laboratory samples. The results are shown in Table II.

TABLE II. Oxide Growth, Commercially Oxidized Parts, Vendor A

Lot No.	Target Intergranular Oxide, μm	Measured Oxide Scale, μm	Measured Intergranular Oxide, μm	Ratio*
1	0.0	0.0	2.5	-
2	2.5	< 1.0	2.5	2.5
3	5.0	1.6	3.0	1.9
3R	5.0	1.5	3.3	2.2
4	10.0	5.8	11.5	2.0
4R	10.0	17.5	19.0	1.1
4RR	10.0	1.0	2.3	2.3
5	15.0	25.0	35.0	1.4
5R	15.0	19.0	24.0	1.3

* Ratio intergranular oxide to oxide scale.

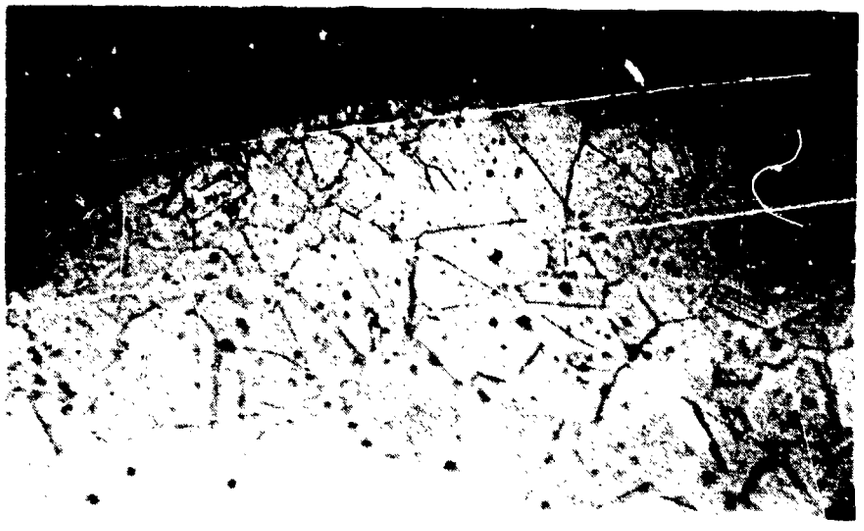
These experiments were made by oxidizing at 1850-1900°F in air, neutral or exothermic atmospheres. Belt speeds of from 5.7-14.0 cm/min were

Lot 1 - Vendor A Oxidized #184 400X 5% Nital

Lot 2 - Vendor A Oxidized #185 400X 5% Nital

Lot 3 - Vendor A Oxidized #186 400X 5% Nital

Fig. 6 Production Furnace Oxidation



Lot 3R - Vendor A Oxidized #278 400X 5% Nital

Lot 4 - Vendor A Oxidized #177 400X 5% Nital

Lot 4R - Vendor A Oxidized #179 400X 5% Nital

Fig. 6 (cont'd) Production Furnace Oxidation

Lot 4RR - Vendor A Oxidized #279 400X 5% Nital

Lot 5 - Vendor A Oxidized #178 400X 5% Nital

Lot 5R - Vendor A Oxidized #180 400X 5% Nital

Fig. 6 (cont'd) Production Furnace Oxidation

used. As can be seen from these results, the target amount of intergranular oxide appears somewhat difficult to achieve. This despite assurances by the supplier that the specified targets would offer no particular difficulty. The indication is that the production pre-oxidizing process contains more uncontrolled variables than is generally realized. From the need for repeated attempts to attain a target oxide quantity, and the general variations from target by most lots, and from some variation within a lot, it appears that much closer pre-oxidation control than is apparently presently practiced will be necessary if imposed criteria are to be met.

As was found in the laboratory oxidation experiments, the production tests also show that intergranular oxidation occurs more rapidly than scale oxide formation during initial stages of oxidation.

In later stages of oxide growth, the ratio of intergranular oxide to scale oxide again approaches 1.5:1, as it did for samples processed in the laboratory.

In comparing the results of production furnace oxidizing with laboratory furnace oxidizing, no critical difference could be noted. The same oxide morphology (scale and intergranular) forms as a function of time, temperature and furnace atmosphere. Interchanging the use of the furnace for de-carburizing, pre-oxidizing and sealing makes closer control imperative since the atmosphere can be set for reducing, neutral, or oxidizing conditions. On occasion, it was evident that a gas-metal reaction (other than oxidation) is taking place during the pre-oxidation process. This effect is shown in Fig. 7, where a distinctive white phase is visible under the scale oxide. This phase is slightly harder (VHN 181) than the parent material (VHN 124), indicating the possibility of a carburizing or nitriding reaction.

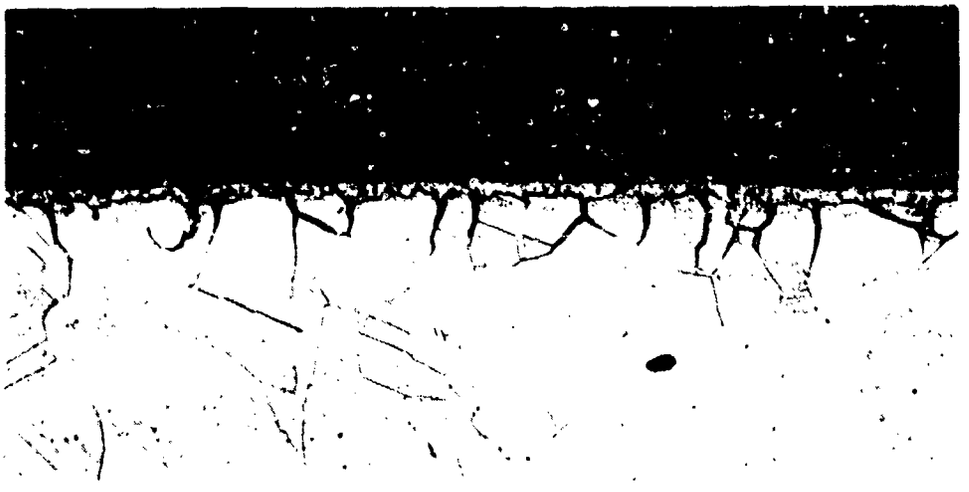


Fig. 7 Second Phase Formation during Commercial Furnace Oxidation. %5 Nital #790 400X

4.0 SEALING

The mechanism by which glass flows and bonds to metal is complex, and still not too well understood. Borom and Pask[4] have studied the role of glass and oxide compositions on bond formation at glass-metal interfaces. They have detailed the behavior of adherence oxides, with emphasis on the effect of these oxides on the glass at the seal interface.

In practice, the glass and metal parts are assembled for sealing in carbon blocks and heated in a slightly oxidizing atmosphere to the working temperature of the glass. As in pre-oxidation firing, the furnace gas cover during sealing can affect the nature and the extent of oxide formation. Also, the oxidizing of the carbon fixtures during sealing can generate reducing gases, and again affect prior oxides.

It should be the objective of sealing to work with a known and consistent pre-oxidized surface condition. The presence of a pre-oxidized surface promotes wetting through a change in glass composition at the seal interface due to solution of the metal oxides in the glass. The oxidized layers facilitate chemical bonding due to glass reactions with the oxide scale, and mechanical bonding due to the surface roughness and enlarged surface area developed by the intergranular oxides. There is considerable latitude available in the degree of oxidation necessary for making glass seals. Generally, with no intergranular oxide, mechanically weak bonds are formed, and with heavy scale, the glass is degraded by oxide saturation. Between these extremes, strong tight seals can be fabricated.

This part of the present study was made to determine the optimum amount of oxidation needed for high reliability matched seals. This was done by procuring sealed TO-5 headers made to the desired interface criteria under production conditions. Finally, a correlation was made between the metallographic criteria and hermeticity test results.

Based on experimental results on oxide growth, orders were placed for different lots of sealed headers to represent product containing residual intergranular oxide spanning a specified thickness range. In addition, samples representing other production lots of sealed packages were obtained from several participating manufacturing facilities. In either case, no attempt was made to dictate firing conditions, except for the requirement that standard commercial practice be used.

4.1 Sealing to Specified Interface Criteria. Vendor A was asked to supply five lots of sealed headers to represent conditions of residual intergranular oxide over the range of from 0.0 - 15.0μm. Residual intergranular oxide is the remnant effect evident in the metal member at a glass-seal interface after the glass has been flowed on a pre-oxidized metal. Shown in Fig. 8 is the transverse section of a glass-metal interface.

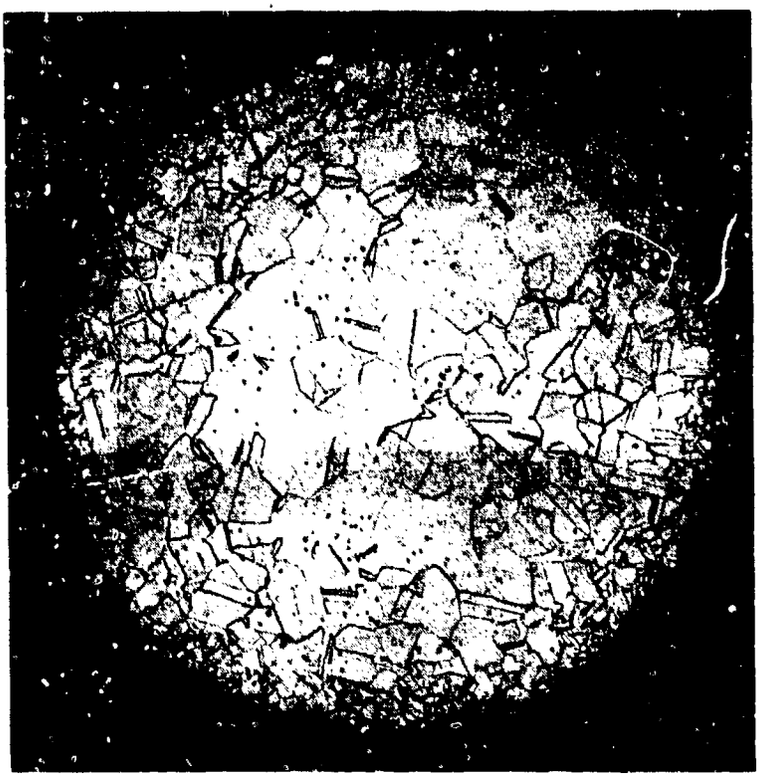


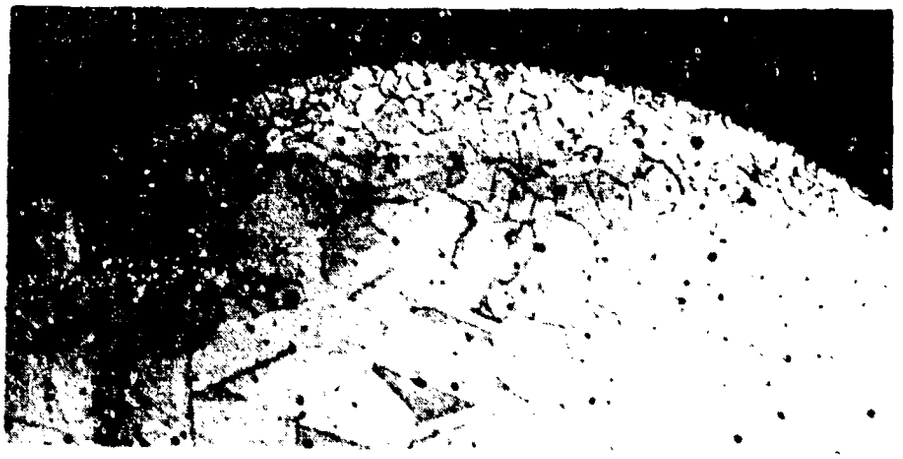
Fig. 8 Transverse Section, Glass-sealed Kovar Lead #791 200X 5% Nital

In a sample such as this, the residual intergranular oxide is measured as an average depth of penetration from the glass-metal interface into the metal member. The measurement is, in this case, 10.0μm which records the depth of the oxidation-affected zone after sealing is completed.

Shown in Table III are the results of the same kind of measurement on five lots of purchased TO-5 headers.

TABLE III. Sealing to Specified Criteria, Vendor A

Lot No.	Specified Intergranular Oxide, μm	Measured Residual Intergranular Oxide, μm (After Sealing)	
		Pin	Eyelet
1	2.5	<2.5	1.9
2	5.0	3.5	3.5
3	7.5	7.5	9.4
4	<1.0	1.2	1.6
5	>7.5	5.5	4.2



Lot 1 Sealed #187 400X 5% Nital
Vendor A <2.5µm Residual Intergranular Oxide

Lot 2 Sealed #188 400X 5% Nital
Vendor A 3.5µm Residual Intergranular Oxide

Lot 3 Sealed #189 400X 5% Nital
Vendor A 7.5µm Residual Intergranular Oxide

Fig. 9 Residual Intergranular Oxide; Lots 1, 2, & 3

Lot 4 Sealed #270 400X 5% Nital
Vendor A 1.2µm Residual Intergranular Oxide

Lot 5 Sealed #266 400X 5% Nital
Vendor A 5.5µm Residual Intergranular Oxide

Fig. 9 (cont'd) Residual Intergranular Oxide; Lots 4 & 5

For each of these lots, three samples were selected at random for metallographic evaluation. The residual intergranular oxide measurements shown in Table III are, therefore, averages from nine (3 headers, 3 leads each) pre-oxidized surfaces. The results indicate that a specified quantity of residual intergranular oxide will probably be difficult to achieve with present controls on commercial pre-oxidizing and sealing processes.

These sections show the nature of the residual intergranular oxide after the glass-metal seal is completed. In each case, the oxide scale has been dissolved by the glass. Upon close examination, Lots 3 and 5 appear to contain the beginning of a halo in the glass due to the presence of sufficient oxide in solution to begin to approach saturation. None of the other lots show this effect.

The effect of the intergranular oxide is to roughen the metal surface at the glass-metal interface. This effect is hardly perceptible at residual intergranular penetration of less than 2.5μm, and is readily perceptible at over 3.5μm of residual intergranular oxide. At 7.5μm, the intergranular oxides approach a magnitude such that surface damage can result in reduced lead integrity (bend fracture or SCC).

From each lot of several hundred sealed headers, a quantity of 75 were processed to complete TO-5 packaging. This included cleaning, plating and cover welding in preparation for testing for hermeticity.

4.2 Hermetic Quality - Control Packages. All the assembled packages in Lots 1, 2 and 3 were tested for hermeticity according to the test plan described in Appendix Item I. The tests were selected for their potential effect on either hermeticity or lead integrity, the most severe condition being MIL-STD-883, Method 1011.2, Cond. C, 100 cycles. As can be seen from the results, no failures occurred in Lot 1, one failure occurred in Lot 2, and one failure occurred in Lot 3. The analysis of these failures showed both to be due to weld seal leaks.

A group of samples from Lots 4 and 5 were tested for hermeticity according to the test plan shown in Appendix Item II. These lots were tested to MIL-STD-883, Method 1011.2, Cond. C prior to further stressing by a thermal pulse. No failure occurred in either lot after Method 1011.2, Cond. C. One failure occurred in Lot 5 after the thermal pulse. This failure was attributed to the presence of a large gas bubble.

Shown in Table IV is a summary of the results due to the metallographic evaluation and the hermeticity testing of packages in Lots 1-5. This comparison shows only one glass seal failure in a total of 345 packages representing different sealing runs for each of 5 lots. Residual intergranular oxide in all metallographically sectioned samples ranged between 1.2 - 9.4μm. The fact that all these samples passed the requirement of MIL-STD-8833, Method 1011.2, Cond. C implies that that level of thermal stress may prove useful for acceptance purposes when package quality is equivalent to these.

4.3 Seals on Purchased Packages. Six lots of purchased packages were obtained from 4 different sources in order to evaluate commercial packages for conformance to hermeticity requirements and to desired metallographic criteria. These packages had been tested for hermeticity according to the test plan shown in Appendix Item III.

TABLE IV. SUMMARY - CONTROL PACKAGES

Lot #	Source	Type Package	No. Units	Hermeticity				Metallography, Lot Acceptance					Failure Mode
				Sample Size	Method	No. Failures	Reject Rate, %	Sample Size	Residual Intergranular Oxide, μm Pin	Residual Intergranular Oxide, μm Eyelet	Gassiness	Interface Foreign Material	
1	Vendor A	TO-5	200	70	1011.2 C 100 cycles	0	0	3	2.5	1.9	<20	None	None
2	Vendor A	TO-5	200	73	1011.2 C 100 cycles	1	1.4	3	3.5	3.5	<20	None	Weld Seal
3	Vendor A	TO-5	200	72	1011.2 C 100 cycles	1	1.4	3	7.5	9.4	>30	None	Weld Seal
4	Vendor A	TO-5	65	65	1011.2 C 15 cycles, thermal pulse	1	1.5	3	1.2	1.6	20	None	Weld Seal
5	Vendor A	TO-5	65	65	1011.2 C 15 cycles, thermal pulse	1	1.5	3	5.5	4.2	>30	None	Large Gas Void (650 x 850μm) Pin - 8.3μm Eyelet - 5.5μm

Figs. 10-17 show the metallographic sections taken of samples obtained from Lots 6-11.

Lot No. 6 parts were experimental by the fabricator. The objective was the introduction of minimum pre-oxidation over deep-etched surfaces. The parts were rejected on the basis of the metallographic evaluation (less than minimum oxide, minimal surface roughening due to etching). Hermeticity test results showed a 6% failure rate. These feed-thrus were made with a 0.38 mm eyelet wall, a 0.51 mm dia. pin and a 1.37 mm glass span (relatively wide).

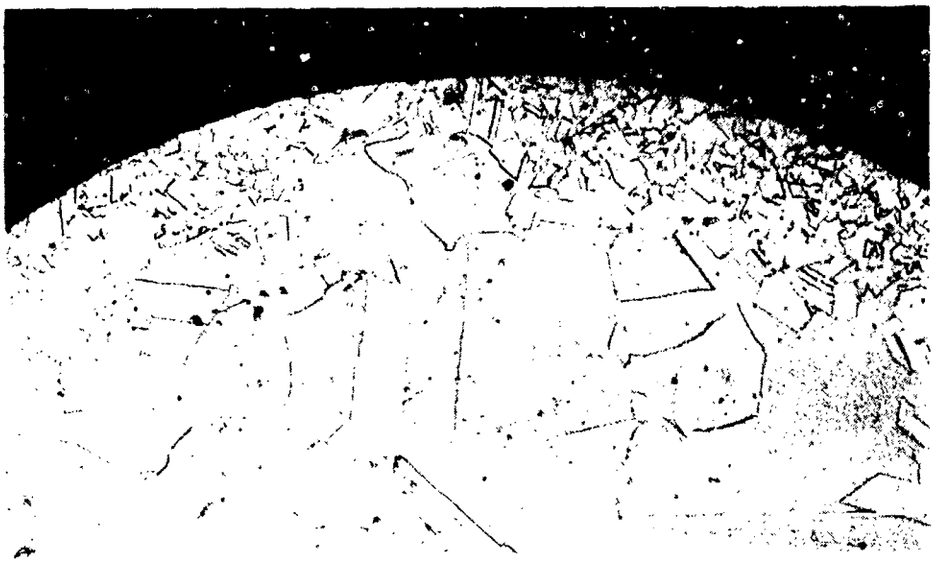
Lot No. 7 parts are representative of large production quantities of a TO-5 type package. The normal rejection rate (due to loss of hermeticity) is 1% for this type of component. This lot experienced a 6% failure rate. Two sets of samples were prepared for metallographic examination. One set was known to have failed hermeticity testing and one set was known to have passed. The leakers show thin (1.75μm) oxide residual and the tight units show acceptable (4.0μm) residual oxide. These units indicate that oxide control on the pin is more critical than it is on the eyelet, also that oxide content varies from pin to eyelet. No other interface problem was noted.

Lot No. 8 parts are also representative of large production quantities of a TO-5 type package. In this case, the normal failure rate is 1%, but this lot passed hermeticity 100%. The residual intergranular oxide is within the desired range; gas quantity, size and distribution are acceptable, and there are no foreign materials at the glass-metal interface.

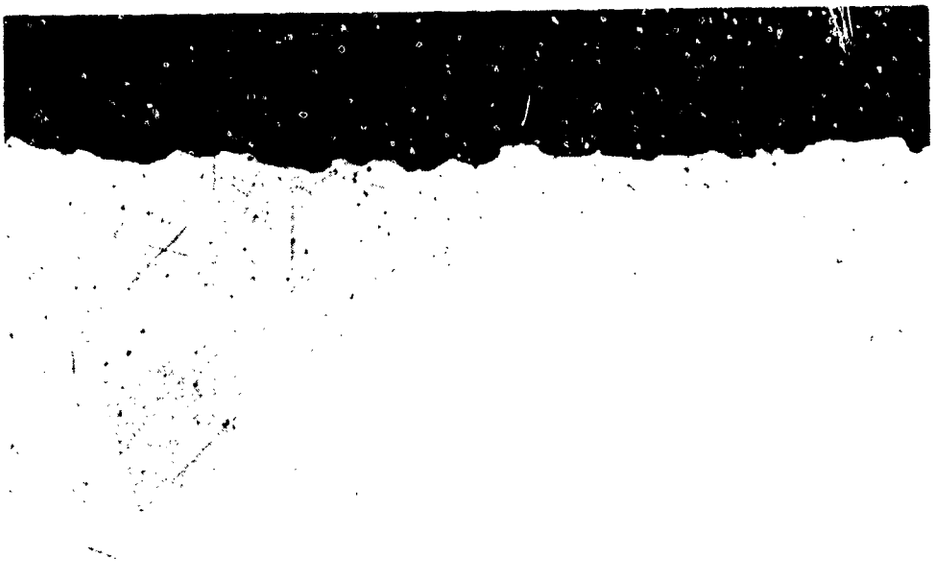
Lots 9 and 10 parts are feed-thru terminals similar to those in Lot No. 6. Lot No. 9 showed a relatively high leak rejection rate due to the effect of excess oxide residuals which caused degradation and cracking of the glass with excessive void formation. Lot No. 10 exhibited a lower failure rate. Most of the metal members were in the acceptable range for residual oxide. Several eyelets were heavily oxidized, resulting in some glass degradation.

Lot No. 11 was experimental by the fabricator. These metal parts were over-oxidized, resulting in glass degradation.

Shown in Table V is a summary of the hermetic and metallographic test results for Lots 6-11. These tests were made on hermetic failures as well as on several acceptable packages from lots with high failure rates. The test results show that: (1) failure rates due to bond separation are lower for units in the range of 1.5 - 6.5μm for residual intergranular oxide, (2) glass-metal bond separation happens more often with residual oxide less than 1.0μm, (3) thin residual oxide appears design sensitive, i.e., higher failure rate when the oxide is thin, if the eyelet is less massive (TO-5 vs. feed-thru), and (4) excess oxide appears to cause leak failures by glass degradation.

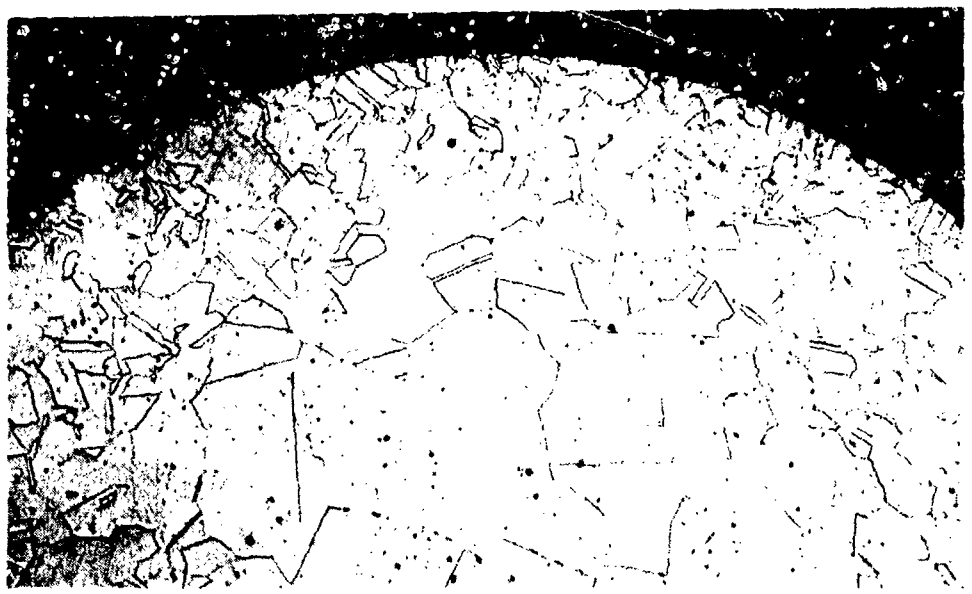


Lot 6 Pin #642 400X 5% Nital
Vendor B <1.0μm Residual Intergranular Oxide,
Pin Separation



Lot 6 Eyelet #641 400X 5% Nital
Vendor B <1.0μm Residual Intergranular Oxide,
Chemical Roughening

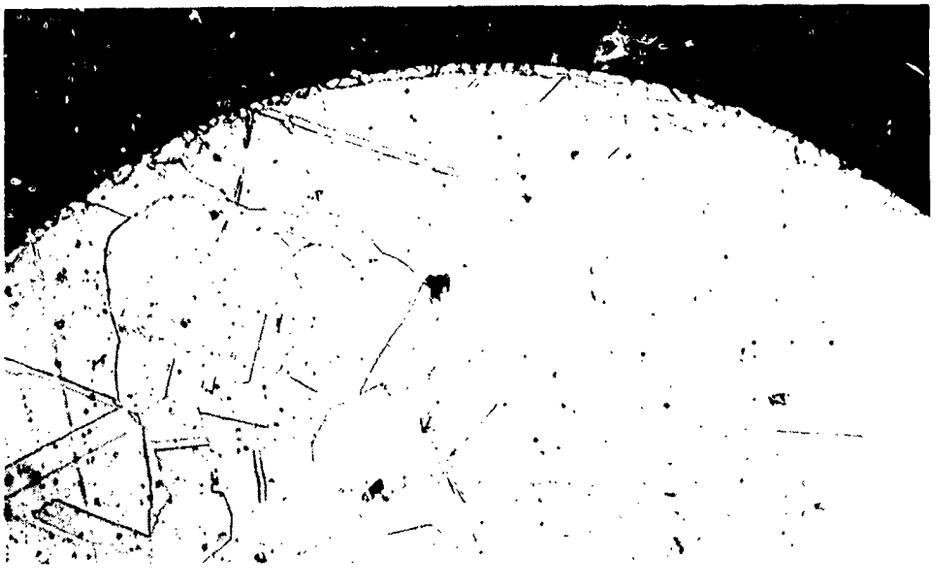
Fig. 10 Lot 6, Hermetic Failure



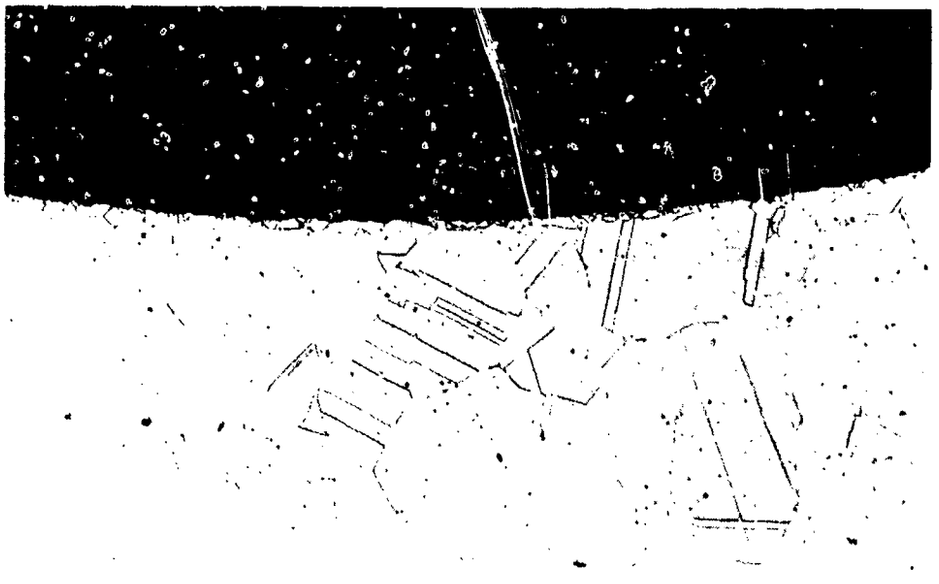
Lot 7 Pin #710 400X 5% Nital
Vendor C 1.75μm Residual Intergranular Oxide

Lot 7 Eyelet #711 40CX 5% Nital
Vendor C <1.0μm Residual Intergranular Oxide

Fig. 11 Lot 7, Hermetic Failure

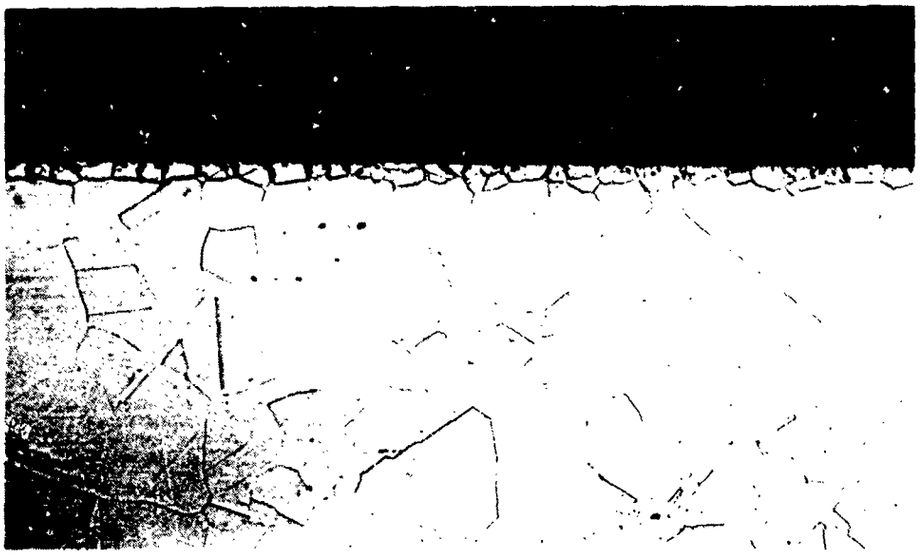


Lot 7 Pin #738 400X 5% Nital
Vendor C 4.0μm Residual Intergranular Oxide

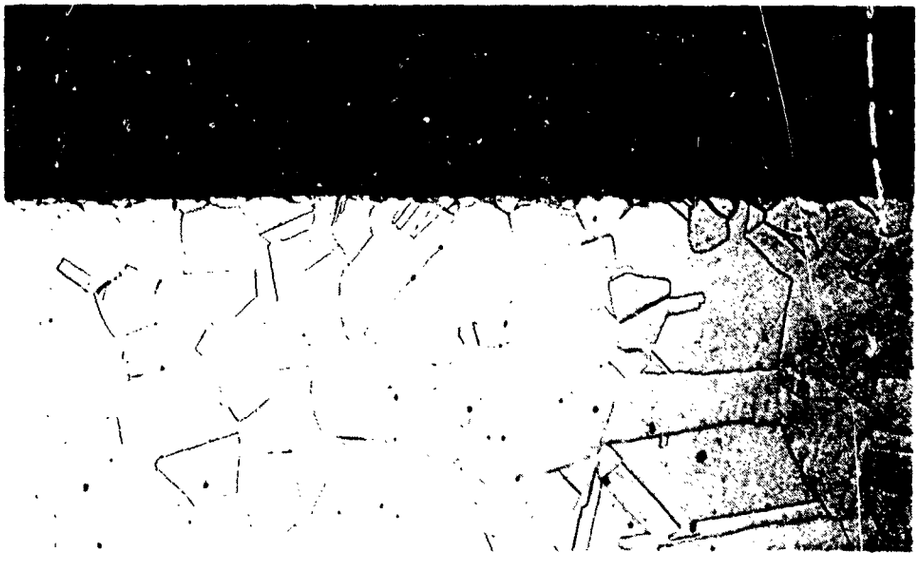


Lot 7 Eyelet #739 400X 5% Nital
Vendor C 1.5μm Residual Intergranular Oxide

Fig. 12 Lot 7, Hermetically Tight Seals

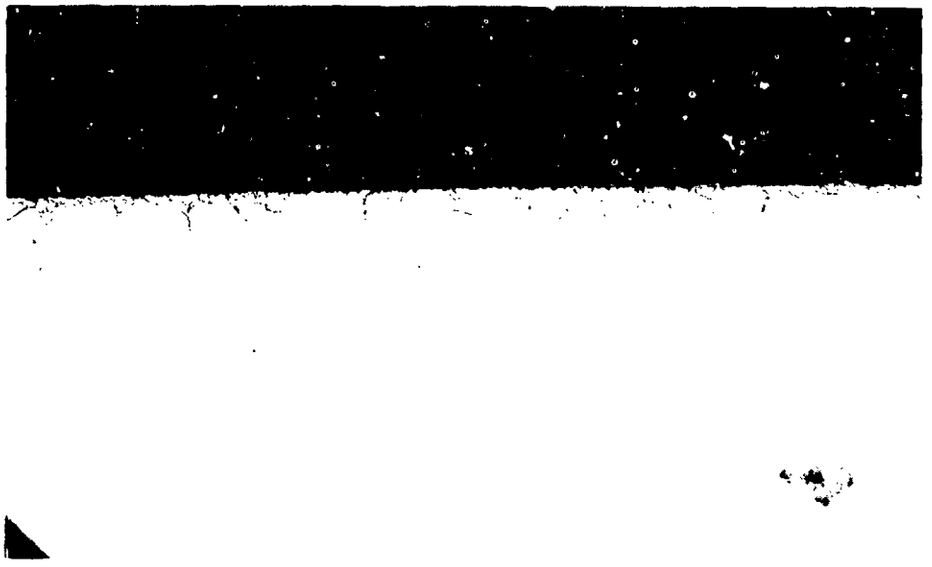


Lot 8 Pin #735 400X 5% Nital
Vendor C 6.5μm Residual Intergranular Oxide,
Longitudinal Section



Lot 8 Eyelet #736 400X 5% Nital
Vendor C 2.5μm Residual Intergranular Oxide,
Longitudinal Section

Fig. 13 Lot 8, Hermetically Tight Seals

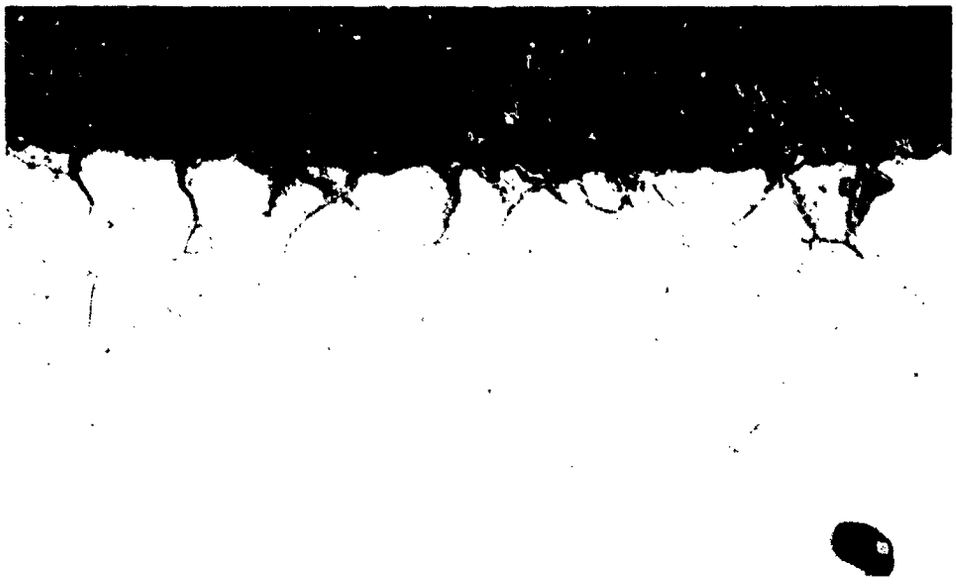


Lot 9	Pin	#611	200X	5% Nital
Vendor D	22.5μm Residual Intergranular Oxide, Glass Saturation, Excess Bubble Formation, Longitudinal Section			

Lot 9	Eyelet	#612	200X	5% Nital
Vendor D	17.5μm Residual Intergranular Oxide, Glass Saturation, Longitudinal Section			

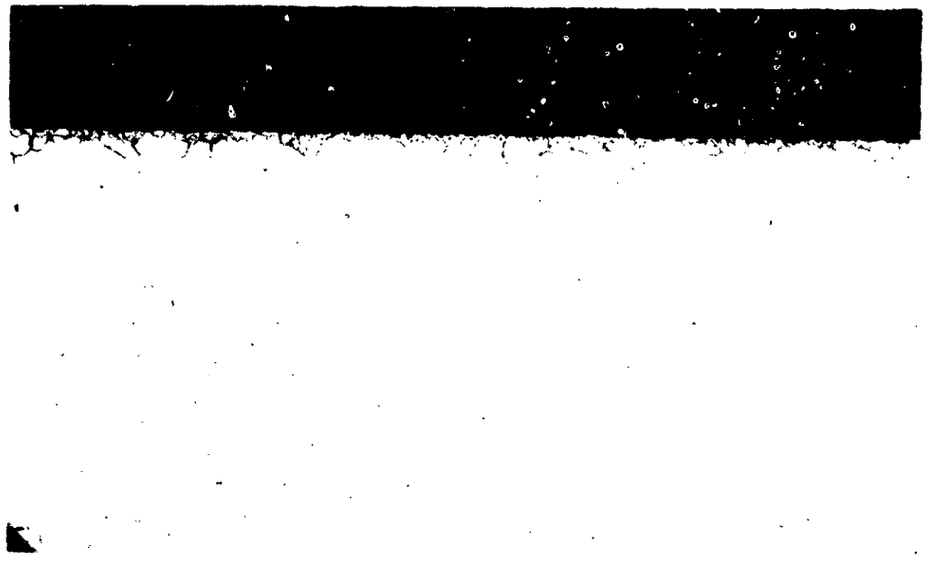
Fig. 14 Lot 9, Hermetic Failure

Lot 10 Pin #619 400X 5% Nital
Vendor D 5.0μm Residual Intergranular Oxide

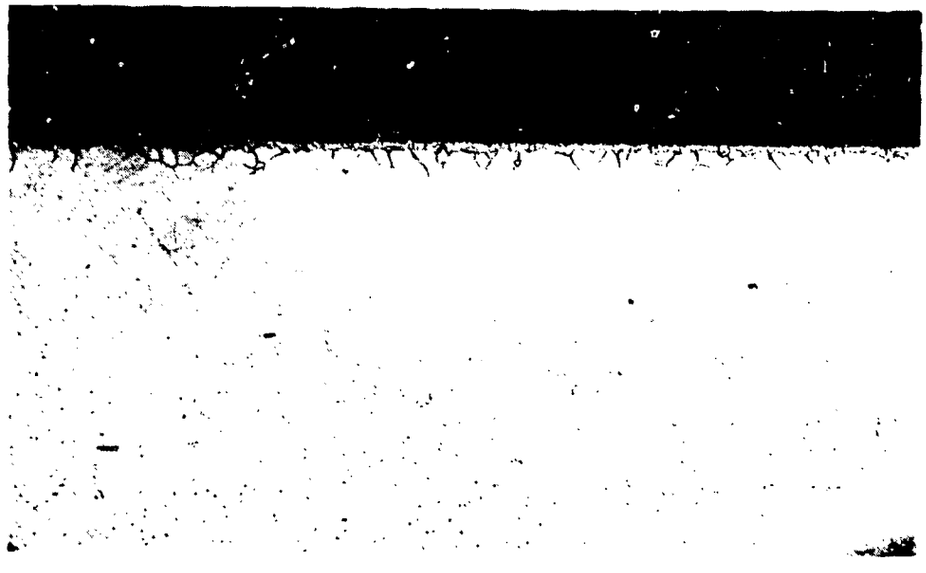


Lot 10 Eyelet #618 400X 5% Nital
Vendor D 25.0μm Residual Intergranular Oxide,
Saturated Glass, Glass Cracked

Fig. 15 Lot 10, Hermetic Failure

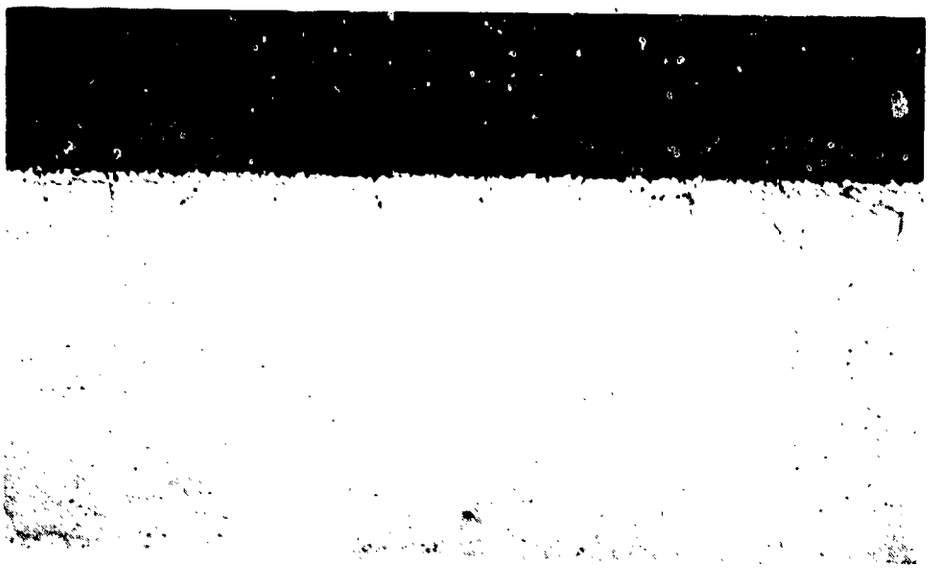


Lot 11 Vendor D	Pin 14.5µm Residual Intergranular Oxide, Saturated Glass, Longitudinal Section	#606	200X	5% Nital



Lot 11 Vendor D	Eyelet 15.7µm Residual Intergranular Oxide, Saturated Glass, Longitudinal Section	#607	200X	5% Nital

Fig. 16 Lot 11, Hermetically Tight Seals



Lot 11 Pin #608 200X 5% Nital
Vendor D 14.5μm Residual Intergranular Oxide,
Saturated Glass, Longitudinal Section

Lot 11 Eyelet #609 200X 5% Nital
Vendor D 17.7μm Residual Intergranular Oxide,
Saturated Glass, Bubble Strings, Longitudinal
Section.

Fig. 17 Lot 11, Hermetic Failure

TABLE V. ANALYSIS OF PURCHASED PACKAGES

Lot #	Source	Type Package	No. Units	Hermeticity				Metallography, Failure Analysis					
				Sample Size	Method	No Fail	Reject Rate, %	Sample Size	Residual Intergranular Oxide, µm Pin	Residual Intergranular Oxide, µm Eyelet	Gassiness	Interface Foreign Material	Failure Mode
6	Vendor B	Feed-thru	150	150	1014, A Torque	9	6.0	2	<1.0	<1.0, etched	0	Void	Poor Pin Bond, Pin Separation
									<1.0	<1.0	0	Void	Poor Pin Bond, Pin Separation
7	Vendor C	TO-5	25000	165	1014, A Cover Weld	10	6.1	2	1.75	<1.0	10%	None	Poor Pin Bond, Hermetic Leaker
									4.0	1.5	10%	None	None
8	Vendor C	TO-5	25000	150	1014, A Cover Weld	0	0.0	1	6.5	2.5	12%	None	None
9	Vendor D	Feed-thru	86	86	1014, A Torque	5	7.0	3	22.7	17.0	5%, Void Alignment	Oxide-Saturated Glass	Gas Voids, Glass Degradation
									22.5	17.0	5%, Void Alignment	Oxide-Saturated Glass	Gas Voids, Glass Degradation
									22.5	17.0	5%, Void Alignment	Oxide-Saturated Glass	Gas Voids, Cracks Glass Degradation
10	Vendor D	Feed-thru	118	118	1014 A Torque	3	2.5	3	4.5	5.0	0	None	Cracked Glass
									5.0	25.0	5%	Oxide-Saturated Glass (Eyelet)	Cracked Glass, Excess Oxide
									6.2	25.0	2%	Oxide-Saturated Glass (Eyelet)	Cracked Glass, Excess Oxide
11	Vendor D	Feed-thru	4	4	1014, A	3	75.0	2	14.5	15.7	2%	Oxide-Saturated Glass (Pin, Eyelet)	None
									17.7	14.5	15%, Void Alignment	Oxide-Saturated Glass (Pin, Eyelet)	Cracked Glass, Pin Separation

5.0 DISCUSSION

There are a number of alternative approaches to glass-metal sealing in practice. We have concerned ourselves primarily with ASTM F15 alloy and Corning 7052 glass during the processing of control-sample experiments. This combination seems to be representative of the hard glass, matched seal where pre-oxidation is an integral, and important part of the sealing process. Although this work applies directly to the Kovar-7052 pair, it is probably valid for other alloys as well as for other glasses.

The results of this work, especially in regard to pre-oxidation, and some of the recommendations that are made do not, however, apply to several other popular sealing methods, the most common being compression seals (including the compression member in combination matched-compression seals), and matched seals made by surface roughening (chemical etching).

There are, of course, a number of choices in the type of glass used in sealing. We have no indication that different glasses affect oxidized Kovar in a widely differing manner. In this study, experimental lots 6-11 behaved as the control lots (1-5) even though other glasses (of unknown composition) may have been used in the production package lots. Therefore, the evaluation criteria for seals using pre-oxidized Kovar should be valid even though glasses other than Corning 7052 are used. This includes ceramic-filled glasses, although more subtle interface reactions occur due to the presence of the ceramic in this type of glass, and more work is required before the proposed test procedure can be used for the control of seals made by the use of ceramic-filled glass.

The furnace atmosphere used in production facilities has significant influence on the manner in which oxidation proceeds up to the time that the seal is effectively complete. This influence results from the use of mixed gases, variations in mix ratios and the use of the same furnaces for degassing, pre-oxidation and sealing by changing gas cover from reducing to oxidizing, as required for a specific purpose. In addition, the present controls on gas generators, and the monitoring of furnace atmospheres for actual conditions are probably not as well developed as they should be for consistency.

This process should be more thoroughly evaluated since important process effects result from uncontrolled gas coverage during pre-oxidation and sealing. The first is the possible radical modification of a pre-oxidized surface by the presence of a reducing gas in the sealing atmosphere, the second is the introduction of surface reactions other than oxidation-reduction, and another is the inhibition of grain growth at the surface. All of these will alter, to some extent, the type and amount of oxide available to the glass when it flows on the metal surface.

The control technique developed here is based on a metallographic evaluation of the metal member for response to a pre-oxidation treatment. The evaluation criteria should apply to any alloy whose oxidation kinetics are similar to those of Kovar. The solubility of the oxides in common glasses will affect the glass member to a greater degree than it will the metal member, and it may reduce seal quality, but this reaction does not affect the oxidation process.

Oxidation is affected by composition, including some impurity elements. We have evidence[5] that impurities, e.g., in Kovar made from secondary metals, may alter oxidation kinetics, although the effect may be more pronounced at the very low levels of oxide quantity. From an evaluation of samples processed to yield a visible intergranular oxide, i.e., over 1.5μm residual intergranular, it appears that impurity levels of materials will have a minimal effect on the oxidation process for Kovar.

The metallographic procedure developed during this program provides a means for monitoring the degree of prior oxidation through an evaluation of seal interfaces after sealing has been completed. At the same time, the metallographic section permits the evaluation of the glass-metal seal quality in several other aspects as well. Gas bubble volume, size and distribution are easily determined through the use of metallography. Also, inspection of the glass-metal interface is possible for defects such as oxide-saturation of the glass, glass-metal separation and foreign materials at the interface.

When comparative evaluations by metallography are made, it appears that the relationship between microstructures and seal quality is as shown in Table VI.

TABLE VI. Observed Effect of Residual Integranular Oxide on Seal Quality

Condition	Residual Intergranular Amount	Observed Effect
Under-oxidation	<1.5μm	poor bond reverse meniscus chemical entrapment leakers (Method 1011.2, Cond. C)
Preferred	2.0-6.5μm	acceptable package to MIL-STD-883B, Method 1011.2, Cond. C
Over-oxidation	>7.0μm	metal surface degradation stress corrosion cracking plating adhesion glass degradation (oxide saturation) excess gassiness excess flow leakers (Method 1011.2, Cond. C)

A determination for residual intergranular oxide appears to be useful for judging the quality of a matched glass-to-metal seal where pre-oxidation is a requisite for sealing. Residual oxides can be detected by metallographic techniques, and the range of 2.0 - 5.5μm seems appropriate for high quality glass seals. Each of the other effects that have been observed, as listed in Table VI, are also detectable by the evaluation of metallographic cross-sections of sealed packages. Acceptance limits for these effects have also been defined in our suggested test procedure (Appendix Item IV). These include volume fraction of bubble formation, bubble size, bubble distribution, glass-metal separation and other interface criteria. The criteria defined here are generally in agreement with those suggested by ISHM in a proposed industry standard[6].

6.0 RECOMMENDATIONS

Due to the wide variation in the nature of commercially fabricated matched glass-to-metal seals, a procedure for tighter control over seal quality is suggested through tighter process control.

The control of the pre-oxidation process is important to improved seal quality, and either the requirement for testing to MIL-STD-883B, Method 1011.2, Cond. C, or the requirement for testing to the metallographic criteria developed in this study are suggested as means for consistently obtaining higher quality seals.

Material combinations other than Kovar-7052 should be evaluated for an examination of the applicability of these test criteria to a broader range of glass-metal seals.

The processes of pre-oxidation and sealing should be studied to determine the suitability of more refined, and automated control techniques for the purpose of obtaining processes capable of better consistency in terms of glass-to-metal seal quality.

7.0 ACKNOWLEDGMENT

The authors appreciate the help of Mr. Donald Blackwood, Mr. Harry Cunningham, Mr. John Droz, Mrs. Mary Guerin and Dr. Hsiu Lin, with special thanks to John McCormick and the Rome Air Development Center for sponsoring the work.

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9.0 APPENDIX

Item I. Lots 1-3 Hermeticity Test Results
Item II. Lots 4-5 Hermeticity Test Results
Item III. Lots 6-11 Hermeticity Test Procedures
Item IV. Proposed Metallographic Test Method

APPENDIX ITEM I

EVALUATION OF TO-5 CAN FOR
E-LAB, SYRACUSE

REPORT NO. QC 2680

OCT. 10, 1973

Report No. QC 2680 October 10, 1978

1. INTRODUCTION AND PURPOSE

The purpose of this task was to determine the seal integrity around the glass beads of three different lots of TO-5 can packages. This task was done per the request of E-Lab, Syracuse.

2. DESCRIPTION OF COMPONENTS

Three lots of TO-5 can devices were provided:

Lot #1 - consisted of 70 samples (1 long lead)
Lot #2 - consisted of 73 samples (2 long leads)
Lot #3 - consisted of 72 samples (3 long leads)

3. SCOPE OF TESTS

All samples were submitted to Visual, Hermeticity, Solderability, Lead Integrity, Shock (Mechanical & Thermal), and Vibration to determine seal integrity of the sealed package as outlined in the Test Plan of Appendix A of this report.

4. CONCLUSIONS

All samples passed Hermetic Seal test at the glass beads even after extended Thermal Shock test.

5. RESULTS AND DISCUSSION

All tests were performed per MIL-STD-883B

1. External Visual - All samples passed.

2. Hermeticity Test - All but two packages tested OK. Two cans leaked at the rim seal. These were removed from the sample.

3. Thermal Shock - Completed.

4. Solderability - All samples passed.
   Note: 7 devices were immersed too far into the solder pot and solder bridged the glass beads.

5. Lead Integrity - All samples passed.

5. RESULTS AND DISCUSSION (Contd)

6. Hermeticity Test - All samples passed.

7. Mechanical Shock - Completed.

8. Two Vibrations - Completed

9. Hermeticity Test - All samples passed.

10. External Visual - Did not reveal any damage or cracks in the glass beads.

Additional testing of Thermal Shock consisting of 15 cycles, 25 cycles, and 60 cycles with hermeticity test after each step did not reveal any leak failures. External visual did not reveal any cracks or damage to the glass beads.

EVALUATION TESTS AND REPORT BY:

D. LLOYD
Sr. Eng. Technician

APPROVED BY:

J. DROZ
Supvr. M & A Lab

APPENDIX A(I)

TEST PLAN

			MIL-STD-883B Method & Conditions	
1.	External Visual		2009.1	
2.	Hermeticity		1014.2	Cond. A_1 & C
3.	Thermal Shock		1011.2	Cond. B
4.	Solderability		2003.2	
5.	Lead Integrity		2004.2	Cond. A, B_1, & B_2
6.	Hermeticity		1014.2	Cond. A_1 & C
7.	Mechanical Shock		2002.2	Cond. B
8.	Vibration Vibration		2005.1 2007.1	Cond. A Cond. A
9.	Hermeticity		1014.2	Cond. A_1 & C
10.	External Visual		2009.1	

Additional Testing:

1.	Thermal Shock	15 cycles	1011.2	Cond. C
2.	Hermeticity		1014.2	Cond. A_1 & C
3.	Thermal Shock	25 cycles	1011.2	Cond. C
4.	Hermeticity		1014.2	Cond. A_1 & C
5.	Thermal Shock	60 cycles	1011.2	Cond. C
6.	Hermeticity		1014.2	Cond. A_1 & C
7.	External Visual		2009.1	

APPENDIX ITEM II
Hermeticity Test Plan
Report for 896680

1. Purpose: These tests were made on two lots of TO-5 packages to determine seal quality.

2. Package Lots: Lot 4 represents 1.2µm residual intergranular oxide on pins, as determined metallographically on 3 samples.

Lot 5 represents 5.5µm residual intergranular oxide on pins, as determined metallographically on 3 samples.

3. Scope:

Pt. I - 15 Pcs Each Lot	MIL-STD-883B Method	Condition
A. External Visual	2009.1	
B. Hermeticity	1014.2	Cond. A, B1, B2
C. Thermal Shock	1011.2	Cond. A
D. Hermeticity	1014.2	Cond. A, B1, B2
E. Thermal Shock	1011.2	Cond. B
F. Hermeticity	1014.2	Cond. A, B1, B2
G. Thermal Shock	1011.2	Cond. C
H. Hermeticity	1014.2	Cond. A, B1, B2

No failures in either lot.

Pt. II - 50 Pcs Each Lot	MIL-STD-883B Method	Condition
A. External Visual	2009.1	
B. Hermeticity	1014.2	Cond. A, B1, B2
C. Thermal Shock	1011.2	Cond. C
D. Hermeticity	1014.2	Cond. A, B1, B2
E. Thermal Pulse *	-	
F. Hermeticity	1014.2	Cond. A, B1, B2

One glass-seal leak occurred in Lot 5 (at F).

* Thermal pulse within 1.25µm of the glass bead by a resistance welding power supply set at 5 watt-seconds and a weld force of 1.4 kg.

4. Metallography: One failure in Lot 5 was found to contain a large (6.5 x 8.5µm) gas void which spanned the glass bead from the lead to the eyelet. Residual intergranular oxide on the pin was 8.3µm and on the eyelet, 5.5µm.

APPENDIX ITEM III
Acceptance Testing - Sealed Packages
Report for 896680

1. Purpose: Six lots of packages were tested for hermeticity by standard incoming acceptance procedures. This was followed by the noted special test. Metallographic comparisons were made for residual oxide.

2. Identification:

A. Lot 6 (Vendor B). Feed-thru (0.5 mm Kovar pin, 1.37 mm glass bead, 0.37 mm wall Kovar eyelet).

150 Pcs	MIL-STD-883B Method	Condition
a. External Visual	2009.1	
b. Hermeticity	1014.2	Cond. A, B1, B2
c. Thermal Stress	1011.2	Cond. A
d. Hermeticity	1014.2	Cond. A, B1, B2
All Pass		
e. Torque	-	
9 seal failures (pin separation)		

B. Lot 7 (Vendor C). TO-5 type (0.45 mm pin, 0.65 mm glass bead, Kovar header.

165 Pcs	MIL-STD-883B Method	Condition
a. External Visual	2009.1	
b. Hermeticity	1014.2	Cond. A, B1, B2
c. Thermal Stress	1011.2	Cond. A
d. Hermeticity	1014.2	Cond. A, B1, B2
e. Cover Weld	-	
10 failures (pin separation)		

Failure analysis shows 1.75µm residual on failed unit, 4.0µm on passed units.

D. Lot 9 (Vendor D). Feed-thru.

86 Pcs	MIL-STD-883B Method	Condition
a. External Visual	2009.1	
b. Hermeticity	1014.2	Cond. A, B1, B2
c. Thermal Stress	1011.2	Cond. A
d. Hermeticity	1014.2	Cond. A, B1, B2
e. Torque	-	
6 failures, cracked glass (oxide saturated), residual to 22.5µm.		

E. Lot 10 (Vendor E). Feed-thru

118 Pcs	MIL-STD-883B Method	Condition
a. External Visual	2009.1	
b. Hermeticity	1014.2	Cond. A, B1, B2
c. Thermal Stress	1011.2	Cond. A
d. Hermeticity	1014.2	Cond. A, B1, B2
e. Torque	-	

3 failures, cracked glass (oxide saturated), residual to 25.0μm.

F. Lot 11 (Vendor E). Feed-thru, experimetal.

4 Pcs	MIL-STD-883B Method	Condition
a. External Visual	2009.1	
b. Hermeticity	1014.2	Cond. A, B1, B2
c. Thermal Stress	1011.2	Cond. A
d. Hermeticity	1014.2	Cond. A, B1, B2
e. Torque	-	

3 failed, cracked glass, pin separation, residual to 17.7μm.

APPENDIX ITEM IV
A Metallographic Test for
Glass-to-Metal Seal Quality

1.0 Purpose: The purpose of this test method is to determine the quality of a matched glass-to-metal seal. This determination is made on the basis of: (1) a measurement of residual intergranular oxide at the glass-metal seal interface, (2) quantity, size and distribution of gas inclusions in the glass, and (3) foreign material or separation at the glass-metal seal interface.

2.0 Apparatus: The apparatus and materials for this test shall include standard metallographic preparation and optical inspection equipment.

3.0 Procedure:

3.1 Sample Size. For glass-metal seal evaluation, a random sample of 3 headers from each lot shall be selected for metallographic sectioning.

3.2 Sections. Samples can be cross-sectioned as shown in Fig. 1. The preferred direction, for this test is Section A-A, normal to the pins. If Section B-B is used, the section plane must be through the center of the pin, and parallel to the pin.

3.3 Sample Preparation. Standard Metallographic techniques are acceptable. Cutting, grinding and polishing should be done with care to avoid cracking the glass and/or introducing artifacts. Polished samples shall be etched with a 5% nitric acid, alcohol solution to accent residual oxides at the glass-metal seal interface.

3.4 Examination. Evaluations are done by the use of a metallurgical (optical) microscope at a minimum of 100X for evaluation of gas voids, and a minimum of 400X for residual intergranular oxide and foreign material.

4.0 Acceptance Criteria:

4.1 Residual Intergranular Oxide. This measurement is taken on glass-metal seals that require a pre-oxidation process for sealing. The average intergranular oxide penetration into the metal member is measured at the glass-metal interface for each metal member making up a matched seal. The limits of allowable intergranular oxide penetration shall be within the range of 2.0 - 6.5μm (0.08-0.26 mil) when a measurement for average depth of penetration is made.

4.2 Undersurface Bubbles (Gas Inclusions). The maximum allowable area of covered (included) bubbles shall be 33% of the total glass area provided these bubbles are not joined in strings or clusters and do not

show or expose the basis metal through the bubbles. Strings of bubbles at the glass-metal interface shall not exceed 20% of the seal length.

Individual or strings of undersurface bubbles shall be no greater than 0.75 mm (0.031") in the longest dimension, and shall occupy no greater than 50% of the distance between any adjacent metal parts.

4.3 Foreign Material or Separation. The presence of foreign material, including excess oxide scale, plating or corrosion product is cause for rejection. Glass-to-metal separation at any point except at the lead exists, shall be rejected. Glass separation at lead exits shall not exceed 20% of the theoretical glass seal thickness when the leads are tested for integrity per MIL-STD-883, Method 2004.1.

5.0 Lot Acceptance. Device lots that fail according to the criteria in para. 4.1 are acceptable if, with the existing LPTD, the lot is tested to, and passes, the requirements of Method 1011.2, Test Condition C; followed by Method 1014.2, Test Condition A or B; followed by Method 1014.2, Test Condition C, D or E.

6.0 Summary. The following shall be specified in the applicable procurement document:

a. Para. 4.1 of this test method applies to matched glass-metal seals with pre-oxidized ASTM F15 alloy.

b. Para. 4.1 of this test method does not apply to matched glass-metal seals with etched metal parts, ceramic filled glass-metal seals, or compression seals.

c. Measurements and examinations at interfaces are made after seals have been fabricated.

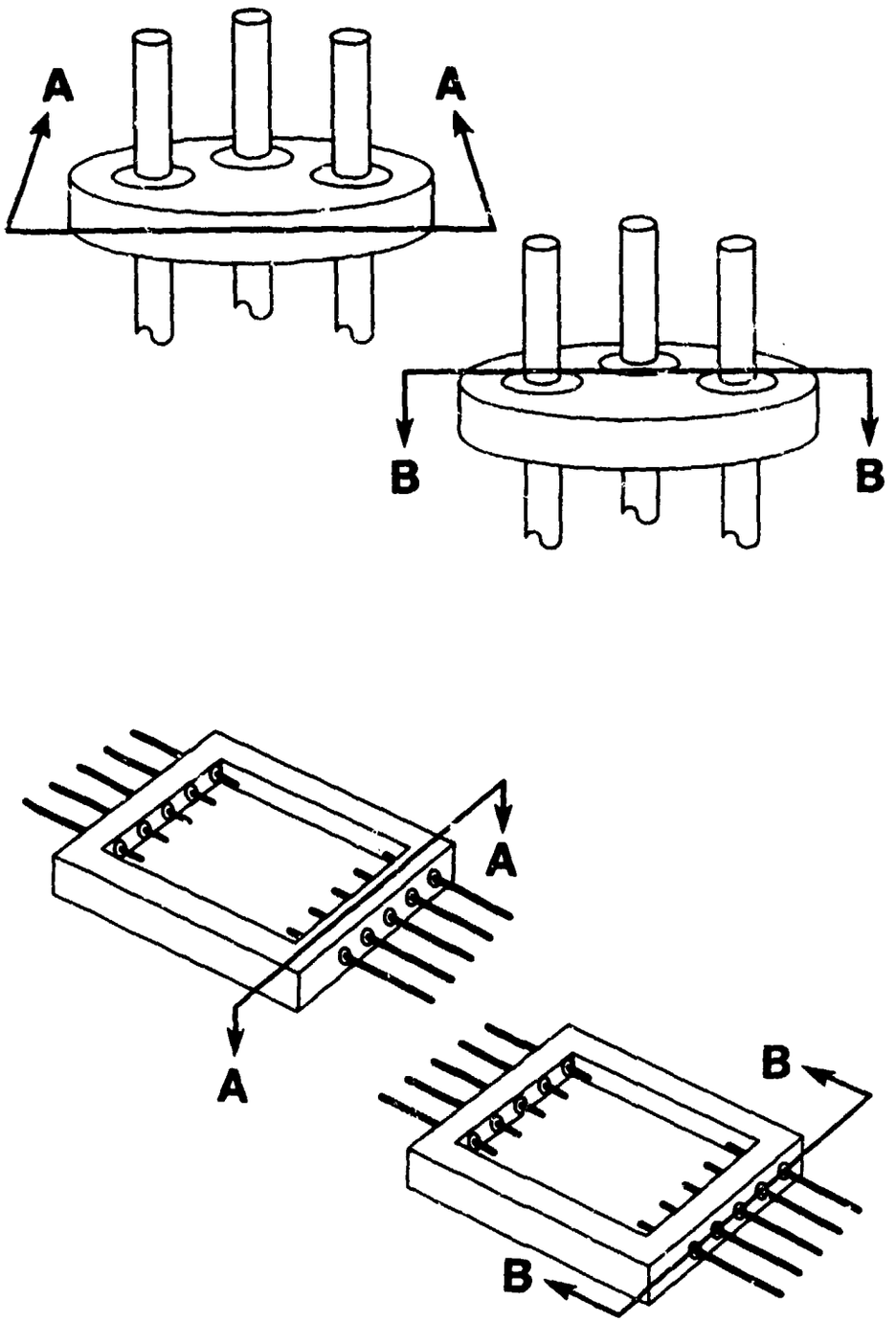

Fig. 1 Sections for Randome and In-Line Lead Orientation